Transformation of the TiNi Alloy Microstructure and the Mechanical Properties Caused by Repeated B2-B19' Martensitic Transformations

Anna Churakova^{1,2} · Dmitry Gunderov^{1,2} · Alexander Lukyanov² · Niklas Nollmann³

Received: 25 March 2015 / Revised: 1 September 2015 / Published online: 12 October 2015
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Abstract The influences of thermal cycling treatment in the temperature range of B2-B19 martensitic transformations (−150 to 150 °C) on the TiNi alloy structure and properties were studied. Different states named the initial coarse-grained (CG) state, the ultrafine-grained (UFG) state after ECAP (with a grain size of 200 nm), the state after ECAP and cold upsetting by 30% were considered. The results show that the microhardness and the strength increase in all the three states. According to the XRD analysis, a more significant increment in the dislocation density, resulting from thermal cycling, is observed in the UFG alloy than in the CG alloy.

KEY WORDS: Thermal cycling; TiNi alloy; Equal-channel angular pressing; Martensitic transformation

1 Introduction

TiNi-based alloys belong to the class of functional materials with a shape memory effect (SME) caused by the thermoelastic martensitic transformations [1–4]. These alloys are widely used as the structural functional materials in engineering and medicine. Processing of ultrafine-grained (UFG) and nanocrystalline (NC) states by severe plastic deformation techniques' service properties makes increase for different metals and alloys, including TiNi-based alloys [5, 6]. The two most popular SPD techniques are high-pressure torsion (HPT) and equal-channel angular

pressing (ECAP) [5]. The ECAP technique enables to produce large-sized samples with a grain size of about 300 nm, and strength and shape memory effect property enhance. The HPT technique enables to produce TiNi samples in the amorphized state, and their subsequent annealing may result in the formation of an NC structure with a grain size starting from 20 nm [6]. Studies have shown that TiNi alloys in the NC state with a grain size of about 50 nm demonstrate the highest service properties [6]. UFG TiNi and NC TiNi are of great interest for practical applications [5, 7, 8].

Thermal cycling (TC) is a technique used for additional enhancement of strength properties in some alloys. The term “thermoelastic transformation” in the strict sense excludes any noticeable irreversible changes. However, in the case of coarse-grained (CG) TiNi, the increase in the dislocation density takes place during multiple cycles of martensitic transformations, which is accompanied by a slight change in the martensitic transformation temperature and some enhancement of the yield stress during mechanical loading. But “phase hardening” resulting from thermal cycling of CG TiNi is not considerable [9]. The materials in the UFG and NC states are characterized by an enhanced specific surface area of the grains (S). The influence of the grain boundaries

Available online at <http://link.springer.com/journal/40195>

✉ Anna Churakova
churakovaa_a@mail.ru

¹ Institute of Molecule and Crystal Physics RAS, 151 pr. Oktyabrya, Ufa 450075, Russia

² Ufa State Aviation Technical University, 12 K. Marx street, Ufa 450000, Russia

³ Institute of Materials Physics, University of Münster, Wilhelm-Klemm Street 10, 48149 Münster, Germany

on defect generation during cyclic martensitic transformations is poorly studied at present. Unlike the majority of other known materials with martensitic transformations (steels, etc.), the temperatures of martensitic transformations in TiNi are close to the room one, that is why thermal cycling does not lead to recrystallization of UFG and NC structures [10]. In this connection, TiNi is an interesting object to study the effects of the UFG and NC states, the grain size and increased area of the grain boundaries on phase hardening resulting from thermal cycling. Grain boundaries as crystalline lattice defects can be the centers for dislocation nucleation during martensitic transformation, and therefore, phase hardening during thermal cycling in UFG TiNi can intensify. On the other hand, in UFG TiNi, martensite plates are limited within a single grain. Thus, in UFG TiNi, the sizes of martensite plates are smaller, which may reduce internal stresses during the growth of martensite plates. In this connection, the intensity of dislocation generation during a cycle of martensitic transformations in a UFG material may decline. It is of special interest to establish the influence of the UFG state of TiNi alloys on the structural changes during thermal cycling through martensitic transformation temperatures, determine the role of the grain boundaries in dislocation generation at phase martensitic transformations, and establish the possibility of enhancement of the mechanical and functional properties of these materials via phase hardening. There are a limited number of publications reporting on the studies of the effect of thermal cycling on UFG TiNi alloys [11–13]. The paper of Tong et al. [11] is devoted mainly to the effect of mechanocycling on the superelastic deformation of ECAP-processed TiNi. The studies of Babicheva et al. [12, 13] mainly deal with the issues of dilatation in UFG TiNi alloys during thermal cycling.

This study presented the effect of the thermal cycling treatment in the temperature range of -150 to 150 °C of TiNi alloy under different states on the structures and properties.

2 Materials and Methods

The studies were carried out on $\text{Ti}_{49.3}\text{Ni}_{50.7}$ (at%) alloy (manufactured by CJSC, Industrial Center MATEX, Moscow) subjected to homogenizing water quenching from 800 °C. The alloy is in the austenite state with a B2 structure (CzCl type) at room temperature.

In order to form a UFG state, quenched cylinder samples ($\varnothing 20$ mm, length 100 mm) were subjected to 8 passes of ECAP using a die-set with the angle of channels' intersection of 120° at 450 °C.

Separated samples after ECAP were subjected to additional deformation by cold upsetting by 30% producing a

UFG state with enhanced dislocation density and shear bands.

Thermal cycling treatment of the samples in different initial states was carried out by cooling to the temperature of liquid nitrogen (-196 °C) and heating to 150 °C, which is a priori lower and higher than the temperatures of direct M_f and reverse A_f martensitic transformations (M_f and A_f). The number of “heating–cooling” thermal cycles was from 0 to 100. The holding time at the heating and cooling temperatures was 3 min.

Calorimetric testing of the material was performed on a Diamond Pyris (Perkin Elmer) differential calorimeter. In order to analyze the martensitic transformation temperatures, differential scanning calorimetry (DSC) was performed on the samples with a diameter of 4.5 mm and a thickness of 0.5 mm in the temperature range from -150 to 150 °C. The heating and cooling rate was 20 °C/min. The characteristic temperatures of the martensitic transformations for this alloy in the quenched state are $M_s = -32$ °C, $M_f = -62$ °C, $A_s = -29$ °C, $A_f = -5$ °C.

The fine structure of the material was studied by a JEM-2100 transmission electron microscope (TEM) at an accelerating voltage of 200 kV. The foils for electron microscopic studies were prepared on a TenuPol-5 twin-jet electropolishing set according to the standard procedure with the help of the electrolyte 10% HClO_4 + 90% $\text{CH}_3(\text{CH}_2)_3\text{OH}$ (90% butanol). The voltage was 50 V. Tensile tests were performed at room temperature with a test speed of 10^{-3} s $^{-1}$ on a tensile machine designed at USATU.

The quantitative studies of the structural parameters were carried out using X-Ray diffraction peak profile analysis (XDPPA). X-ray diffraction patterns were obtained on a Rigaku Ultima IV diffractometer in the range of 2θ from 30° to 120° . The dislocation density and other parameters were determined via the procedure described in [14, 15].

3 Results and Discussion

In the initial CG state at the room temperature, the $\text{Ti}_{49.3}\text{Ni}_{50.7}$ alloy has an austenitic structure with a grain size of about (50 ± 5) μm according to optical metallography analysis (Fig. 1a). Optical metallography does not allow performing precision estimation of the structural changes after thermal cycling (Fig. 1b). According to the TEM, dislocation-free grain boundaries are observed in the CG state (Fig. 1c). After thermal cycling with a number of cycles equal to 20, the dislocations pile up near the grain boundaries due to phase hardening (Fig. 1d).

The subsequent increase in the number of the thermal cycles to 100 has apparently resulted in an additional

Fig. 1 Structures of $\text{Ti}_{49.3}\text{Ni}_{50.7}$ in different states: **a** CG, **b** CG + TC (OM), **c** CG (TEM), **d** CG + TC ($n = 20$), **e** CG + TC ($n = 50$), **f** CG + TC ($n = 100$)

increase in the dislocation density in the grains body, near the grain boundaries and triple joints (Fig. 1 e, f).

After ECAP processing, a UFG structure with a grain size of about 200 nm is formed in TiNi (Fig. 2a). As the result of thermal cycling increase to 20, 50 and 100, the dislocation density increases apparently as shown in (Fig. 2b, c, d).

Cold upsetting by 30% of an ECAP-processed sample usually leads to the formation of deformation bands and a

significant increase in the dislocation density in the structure. The bandwidth and the size of structural elements are about 50 nm (Fig. 3a). Thermal cycling leads to the increase in the dislocation density in some separated structure areas (Fig. 3b); however, the TEM method did not show quantitative changes in the structure with an increasing number of cycles from 50 to 100 (Fig. 3c, d).

As is reasonably expected (Table 1), the values of peak broadening and dislocation density in UFG alloy are

Fig. 2 Microstructures of $\text{Ti}_{49.3}\text{Ni}_{50.7}$ in the UFG state: **a** UFG, **b** UFG + TC ($n = 20$), **c** UFG + TC ($n = 50$), **d** UFG + TC ($n = 100$)

notably higher, and the CSR size is markedly smaller, than those in CG TiNi. In the state UFG + upsetting (Table 1), the values of peak broadening and dislocation density are visibly higher than those in the UFG alloy.

The values of peak broadening and dislocation density in the CG TiNi alloy slightly grow, while the CSR dimensions slightly decrease, as a result of thermal cycling. A similar increment in peak broadening has been observed earlier, as a result of the thermal cycling of the CG $\text{TiNi}_{50.7}$ alloy [17].

The analysis of the XRD data (Table 1) demonstrates that in the UFG alloy the values of peak broadening grow more significantly as a result of thermal cycling than those in the CG TiNi alloy. The increment in the dislocation density, resulting from thermal cycling, was $\Delta \rho \approx 1 \times 10^{15} \text{ m}^{-2}$ in the CG alloy and $\Delta \rho = 3.7 \times 10^{15} \text{ m}^{-2}$ in the UFG alloy. An even larger increment in the dislocation density, resulting from thermal cycling, is observed in the UFG + upsetting alloy – $\Delta \rho \approx 6.2 \times 10^{15} \text{ m}^{-2}$. Therefore, a conclusion can be made that the high density of the grain boundaries and dislocations, both for the states of UFG and UFG + upsetting, contributes to the intensification of phase hardening (dislocation density enhancement) during thermal cycling (Fig. 4).

The dimensions of coherent scattering regions in CG TiNi significantly decrease as a result of thermal cycling.

In the UFG and UFG + upsetting alloys, the dimensions of CSR also decrease as a result of thermal cycling, but not so much as in CG TiNi. Table 2 shows the results of the studies on the martensitic transformation temperatures, performed by DSC.

The analysis of calorimetric data allows making a conclusion that the thermal cycling results in an increase in the martensitic transformation temperatures, especially M_s and M_f . The most significant increases in the temperatures M_s and M_f are observed for the UFG state (by 50 °C and 35 °C, respectively), whereas in the CG state the temperatures M_s and M_f increase approximately to 25 °C. In the UFG alloy, as a result of TC, the A_f temperature also increases significantly (29 °C). At the same time, in the CG alloy, the temperatures A_s , A_f change insignificantly as a result of TC. It has been shown earlier in some papers [4, 16] that during thermal cycling of CG TiNi the martensitic transformation temperatures M_s and M_f slightly decrease. According to our investigations, the martensitic transformation temperature increases in TiNi as a result of TC. This can be conditioned by alloy aging due to the used thermal cycling regimes or by intermediate R-transformation. This phenomenon requires additional studies.

Fig. 3 Structures of $\text{Ti}_{49.3}\text{Ni}_{50.7}$: **a** ECAP + cold upsetting, **b** ECAP + cold upsetting and TC ($n = 20$), **c** ECAP + cold upsetting and TC ($n = 50$), **d** ECAP + cold upsetting and TC ($n = 100$)

Table 1 Parameters of the $\text{Ti}_{49.3}\text{Ni}_{50.7}$ structure according to X-ray analysis

State	FWHM (110)	CSR (nm)	E (%)	$\rho \times 10^{15} (\text{m}^{-2})$
CG	0.282	158	0.122	0.8
CG + TC	0.347	49	0.221	1.8
Δ	0.065	109	0.099	1
UFG	0.327	43	0.250	5.8
UFG + TC	0.440	40	0.465	9.6
Δ	0.113	3	0.215	3.8
UFG + upsetting	0.96	31	0.647	16
UFG + upsetting + TC	1.13	27	0.854	22
Δ	0.17	4	0.207	6.2

FWHM is X-ray peak broadening; CSR is coherent scattering regions, nm; E is root-mean-square microdistortions, %; $\rho \times 10^{15}$ is dislocation density, m^{-2} ; Δ is the difference in the corresponding parameters prior to and after thermal cycling

The DSC technique did not allow determining the martensitic transformation temperatures in the states UFG + upsetting and UFG + upsetting + TC. However, TC leads to growth of the microhardness and the dislocation density in the UFG + upsetting state. Consequently, the martensitic transformations take place in this state as well. Perhaps, in the UFG + upsetting state, the sample has microstructure inhomogeneity. Therefore, in different

areas of the sample, transformations take place at different temperatures, and as a result, separated transformation peaks are not determined by DSC.

The microhardness H_V of the $\text{Ti}_{49.3}\text{Ni}_{50.7}$ alloy in all the studied states increases as a result of thermal cycling as compared to the initial state (Fig. 5).

The microhardness H_V increases monotonously at the CG and UFG states with the number of cycles from 0 to

Fig. 4 X-ray diffraction patterns of the alloy $\text{Ti}_{49.3}\text{Ni}_{50.7}$: 1 CG, 2 CG + TC; 3 UFG, 4 UFG + TC; 5 UFG + cold upsetting, 6 UFG + cold upsetting + TC

Table 2 Characteristic temperatures of martensitic transformations

State	M_s	M_f	A_s	A_f	$M_s - M_f$	$A_s - A_f$
CG	-32	-62	-29	-5	30	24
CG + TC*	-6	-41	-39	-2	35	37
UFG	-23	-47	-11	3	24	14
UFG + TC*	27	-12	-3	32	39	35

* Thermal cycling $n = 100$. M_s is martensitic transformation start temperature; M_f is martensitic transformation finish temperature; A_s is reverse transformation start temperature; A_f is reverse transformation finish temperature

100. As for the UFG + upsetting state, the microhardness grows intensively during the first 20 cycles and is followed by saturation with a further increase in the number of cycles to 100. But the microhardness increment during the thermal cycling of the UFG state after upsetting is higher than that in the UFG and CG states (Fig. 5, Table 3).

As shown in Fig. 6, the ultimate tensile strength σ_B of $\text{Ti}_{49.3}\text{Ni}_{50.7}$ in the CG state is 820 MPa and increases to 900 MPa as a result of thermal cycling due to phase hardening. Deformation-induced martensitic transformation as a phase pseudo-yield plateau typically of the CG state is observed on the plots. The values of the stress of deformation-induced martensitic transformation (phase yield stress σ_m) also grow as a result of thermal cycling, from 440 to 620 MPa. This is accounted by the fact that the dislocations in the austenite phase, which formed as a result of thermal cycling, hamper deformation-induced martensitic transformation. Note should be made that the stress of dislocation flow (YS) of the CG alloy is very close to the value of σ_m . In this connection, the YS value of the CG alloy and its increment resulting from TC are difficult to be determined precisely. However, according to our

Fig. 5 Microhardness plots under different states (1 CG, 2 UFG, 3 UFG + cold upsetting)

estimation, the YS of the CG alloy has increased by 100 MPa as a result of TC (note should be made that in the paper [17] an approximately proportional increment was observed in the YS of the CG $\text{TiNi}_{50.7}$ alloy as a result of TC, $n = 50$), but the ductility of the CG alloy was reduced from 36 to 31% a result of thermal cycling.

The ultimate tensile strength value (1310 MPa) and the yield stress value (1130 MPa) of the UFG alloy are significantly higher than those of the CG alloy (820 and 520 MPa, respectively). At the same time, the phase yield stress values (σ_m) of the UFG alloy and the CG alloy are almost identical (450 and 440 MPa, respectively). Thus, the formation of a UFG structure with an increased density of the grain boundaries efficiently increases the yield stress of the alloy, but has little effect on the phase yield stress values. Thermal cycling insignificantly increases the UTS and YS of the UFG alloy to 1360 MPa and 1200 MPa (Table 3). The increments in the ultimate tensile strength and YS of the UFG alloy, resulting from thermal cycling, are even somewhat smaller than those in the CG alloy. At the same time, in the UFG alloy, the dislocation density grows more significantly, as a result of thermal cycling, than that in CG TiNi (Table 3). Perhaps, the small increment in the UTS and YS of the UFG alloy, resulting from thermal cycling, can be accounted for by the fact that the UFG alloy is already in a strengthened state. The phase yield stress values of the UFG alloy also grow as a result of thermal cycling (Table 3).

In the UFG + upsetting state, the ultimate tensile strength was about 1750 MPa, which is noticeably higher than that in the UFG state. It should be noted that the curve rate for the state with a high density of defects differs from those of the CG and UFG states. There is no phase pseudo-yield plateau on the curves for the UFG + upsetting alloy,

Table 3 Results of mechanical tensile tests of $\text{Ti}_{49.3}\text{Ni}_{50.7}$ in different states

State	UTS (MPa)	σ_m (MPa)	YS (MPa)	δ (%)	HV(MPa)
CG	820 ± 20	440 ± 10	520 ± 15	36 ± 2	2695 ± 70
CG + TC*	900 ± 15	620 ± 10	620 ± 10	31 ± 2	3150 ± 75
UFG	1310 ± 20	450 ± 10	1130 ± 10	29 ± 1	3550 ± 90
UFG + TC*	1360 ± 10	530 ± 15	1200 ± 15	31 ± 1	4070 ± 80
UFG + upsetting	1750 ± 20	–	1410 ± 20	22 ± 2	4860 ± 120
UFG + upsetting + TC*	1910 ± 25	–	1550 ± 15	26 ± 3	5425 ± 95

* Thermal cycling $n = 20$ **Fig. 6** Stress–strain curves for $\text{Ti}_{49.3}\text{Ni}_{50.7}$: 1 CG, 2 CG + TC; 3 UFG, 4 UFG + TC; 5 UFG + cold upsetting, 6 UFG + cold upsetting + TC

which indicates the blocking of deformation-induced martensitic transformation. But it is more probable that in the UFG + upsetting alloy, martensitic transformations occur at different loads, and as a consequence, a pseudo-yield plateau is not observed on the tensile curve. The absence of phase yield in the deformed TiNi has been noted earlier, in particular in [6]. The thermal cycling is enabled to achieve an increase in the ultimate tensile strength by 200 MPa, up to 1960 MPa.

Hence, the treatment by thermal cycling of the UFG + upsetting alloy has allowed achieving the highest values of YS, UTS and microhardness in the $\text{Ti}_{49.3}\text{Ni}_{50.7}$ alloy. Here, the elongation in this state remains rather good.

Thus, according to the data from XRD analysis, in the UFG and UFG + upsetting alloys, the dislocation density grows more significantly, as a result of thermal cycling, than that in CG TiNi (Table 1). Consequently, the grain boundaries, the density of which is higher in UFG materials, are additional dislocation generation areas at diffusionless martensitic phase transformations.

On the basis of the obtained data, the following simplest evaluations of the effect of the grain boundaries on

dislocation generation during cycles of martensitic transformations can be proposed.

Let us designate the dislocation density growth in the CG material after thermal cycling as $\rho_{\text{CG}\Delta}$, and the dislocation density growth in the UFG material as $\rho_{\text{UFG}\Delta}$.

In the simplest approximation, if one accepts that the grains have the same size and a cubic shape, the area of the intergrain boundaries (S) in a volume unit of a polycrystalline material is given by equation:

$$S = 3/D, \quad (1)$$

where D is the grain size.

In CG TiNi at D_1 (the grain size in the CG material) = 50 μm , $S = 60 \text{ mm}^2/\text{mm}^3$; in UFG TiNi $D_2 = 0.3 \mu\text{m}$, $S = 10^4 \text{ mm}^2/\text{mm}^3$.

Let us designate the density of dislocations generated during thermal cycling in the material volume as $\rho_{\Delta\text{V}}$.

It shall be accepted that the density of dislocations generated in the grain boundaries during thermal cycling is directly proportional to the grain area:

$$\rho_{\Delta\text{G}} = K_1 \times S, \quad (2)$$

where K_1 is a certain coefficient and S is the area of the grain boundaries.

Then, the total density of dislocations generated in a volume unit during thermal cycling is as follows:

$$\rho_{\Delta} = \rho_{\Delta\text{V}} + K_1 \times S. \quad (3)$$

Therefore, we obtain a system of equations:

$$\rho_{\text{CG}\Delta} = \rho_{\Delta 0} + K_1 \times 3/D_1, \quad (4)$$

$$\rho_{\text{UFG}\Delta} = \rho_{\Delta 0} + K_1 \times 3/D_2.$$

According to the data from X-ray structural analysis (Table 1), $\rho_{\text{CG}\Delta} = 1.07 \times 10^{15} \text{ m}^{-2}$, $\rho_{\text{UFG}\Delta} = 3.71 \times 10^{15} \text{ m}^{-2}$.

Using the available experimental data for $\rho_{\text{CG}\Delta}$, $\rho_{\text{UFG}\Delta}$, D_1 and D_2 , we shall obtain the following results.

The density of dislocations generated in a volume unit of TiNi at one hundred cycles of martensitic transformations is $\rho_{\Delta v} \approx 1.0583 \times 10^{15} \text{ m}^{-2}$.

The coefficient reflecting the quantity of dislocations generated by a surface area unit in a material volume unit at 100 cycles of martensitic transformations is $K_1 = 1.8 \times 10^8 \text{ m}^{-1}$.

4 Conclusions

- (1) According to TEM and OM, the Ti_{49.3}Ni_{50.7} alloy has a structure with a grain size of 50 μm in the initial CG state and a grain size of 200 nm in the UFG state, and the size of structural elements equals 50 nm in the UFG + upsetting state.
- (2) According to TEM, as a result of thermal cycling, an increase in the dislocation density is observed in the vicinity of the grain boundaries and triple junctions in the CG and UFG states.
- (3) The calorimetric data show that the thermal cycling results in an increase in the martensitic transformation temperatures, especially M_s and M_f .
- (4) According to the X-ray analysis, a more significant increment of the dislocation density is observed in the UFG alloy than in the CG one. The highest dislocation density increment is observed as a result of thermal cycling in the UFG + upsetting state—a state with the most refined structure. Thus, the grain boundaries, the density of which is higher in UFG materials, are additional dislocation generation areas at diffusionless martensitic phase transformations.
- (5) As a result of thermal cycling, the microhardness of the alloy in the CG and UFG states increases. The highest microhardness increment is observed a result of thermal cycling after UFG + upsetting. As for the UFG + upsetting state, the microhardness grows intensively during the first 20 cycles and is followed by saturation with a further increase in the number of cycles to 100.
- (6) A set of enhanced mechanical properties of the strength, yield stress and ductility is achieved as a

result of the combined treatment that includes ECAP, upsetting and thermal cycling.

Acknowledgments The author Anna Churakova acknowledges the support for XRD investigation (the Alexander von Humboldt Foundation through the Group Linkage Project Fokoop-DEU/1052606). The author Dmitry Gunderov acknowledges the support for TEM investigation (the RFBR Project No. 15-08-04784). The author Alexander Lukyanov acknowledges the support for ECAP processing (the Russian Science Foundation through Project No. 14-12-00138).

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